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पशु आहार तथा भरण सामग्री – कैल्शियम ज्ञात करना

भाग 2 आण्विक अवशोषण स्पेक्ट्रोमापी

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS — DETERMINATION OF CALCIUM

PART 2 ATOMIC ABSORPTION SPECTROMETRIC METHOD

UDC 636·085 : 543·422 [546·41]

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

August 1992

Price Group 2

Livestock Feeds Sectional Committee, FAD 5

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Livestock Feeds Sectional Committee had been approved by the Food and Agriculture Division Council.

IS 7874 Methods of test for animal feeds and feeding stuffs is available in three parts:

Part 1 General method
Part 2 Mineral and trace elements
Part 3 Microbiological test methods

A test method for determination of calcium has been prescribed in **5** of IS 7874 (Part 2) : 1975. During usage of this test method, need has been felt to revise it, since it has reported to give low recovery.

This Indian Standard which replaces the contents given under **5** of IS 7874 (Part 2) : 1975 is being published in three parts:

Part 1 Titrimetric method
Part 2 Atomic absorption spectrometric method

This standard (Part 2) covers the atomic absorption spectrometric method for determination of calcium in animal feeds and feeding stuffs — is based on ISO 6490/2 : 1983 Animal feeding stuffs — Determination of calcium content Part 2 : Atomic absorption spectrometric method and is technically equivalent to it. This method is recommended for determination of very low calcium contents (lower than 0·1 percent).

Part 1 of the standard covers the titrimetric method for determination of calcium in animal feeds and feeding stuffs. It is based on ISO 6490/1 : 1985. Animal feeding stuffs — Determination of calcium content: Part 1 Titrimetric method is technically equivalent to it. This method is applicable to all animal feeding stuffs having calcium content greater than 1 g/kg.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ANIMAL FEEDS AND FEEDING STUFFS — DETERMINATION OF CALCIUM

PART 2 ATOMIC ABSORPTION SPECTROMETRIC METHOD

1 SCOPE

This Indian Standard (Part 2) specifies an atomic absorption spectrometric method for the determination of the calcium content of animal feeding stuffs.

The limit of detection is in the region of 10 mg/kg.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
460 (Part 1) : 1985	Specification for test sieves: Part 1 Wire cloth test sieve
1070 : 1992	Reagent grade water — Specification (*third revision*)
13433 (Part 1) : 1992	Methods of test for animal feeds and feeding stuffs: Part 1 Titrimetric method

3 PRINCIPLE

Following any necessary destruction of organic matter by incineration of a test portion, dissolution of the calcium by treatment with hydrochloric acid and dilution of the solution obtained, in the presence of lanthanum which is used as a spectral buffer. Determination of the calcium content by atomic absorption spectrometry.

4 REAGENTS

All reagents shall be of recognized analytical quality and the water used shall be double-distilled, deionized and distilled or double deionized water.

4.1 Hydrochloric Acid Concentrated — (P_{20} = 1.18 to 1.19 g/ml)

4.2 Hydrochloric Acid — 6 mol/l solution.

4.3 Lanthanum Chloride — solution prepared as follows:

In a 1 000 ml one-mark volumetric flask. dissolve 25 g of lanthanum oxide of low calcium content in 75 ml of the hydrochloric acid (**4.1**). Following reaction, allow to cool, add a little water, agitate, dilute the mark with water and mix.

4.4 Calcium — standard solution corresponding to 40 mg/l.

4.4.1 *Stock Solution* — corresponding to 1 g/l.

Weigh 2.497 g of calcium carbonate which has been previously dried at 105°C for 1 h. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, rinsing with about 100 ml of water. Add 50 ml of the hydrochloric acid (**4.1**) to dissolve the carbonate, dilute to the mark with water and mix.

4.4.2 *Standard Solution* — corresponding to 40 mg/l.

By means of a pipette, transfer 10 ml of the stock solution (**4.3.1**) into a 250 ml one-mark volumetric flask. Add several drops of the hydrochloric acid (**4.1**), dilute to the mark with water and mix.

5 APPARATUS

Usual laboratory equipment, and in particular the following.

5.1 Electrical Heated Muffle Furnace — preferably of silica, capable of being controlled at 550 ± 10°C.

5.2 Incineration Dishes — of platinum, or, failing this, silica or porcelain.

5.3 Atomic Absorption Spectrometer — equipped to determine calcium, using an air/acetylene flame.

5.4 Ashless Filter Paper

5.5 Beaker — of 250 ml capacity.

5.6 Volumetric Flask — of 100 and 250 ml capacity.

5.7 Pipettes — to deliver 5, 10, 15, 20 and 25 ml.

5.8 Sand Bath or Hot Plate — capable of being controlled at approximately 150°C.

5.9 Analytical Balance

6 PROCEDURE

6.1 Preparation of the Test Sample

Grind about 100 g of the sample so that it passes through 1·00 mm IS Test sieve [*see* IS 460 (Part 1) : 1985]. Transfer this prepared sample to a well stoppered glass bottle.

6.2 Test Portion

6.2.1 *Sample Containing Organic Matter*

Weigh to the nearest 1 mg, 1 to 5 g of the test sample, according to the expected calcium content, into an incineration dish (**4.2**).

6.2.2 *Sample not Containing Organic Matter*

Weigh to the nearest 1 mg, 1 to 5 g of the test sample, according to the expected calcium content, into a 250 ml beaker (**5.5**).

6.3 Preparation of the Test Solution

6.3.1 *Incineration and Rendering the Silica Insoluble*

(Only in the case of samples containing organic matter).

Place the incineration dish in the muffle furnace (**5.1**) while cold, close the furnace and gradually increase the temperature so as to reach 550 ± 10°C in approximately 1 h 30 min. Maintain this temperature until the ash is free of carbon particles (if necessary maintain this temperature for 16 h; that is overnight), then remove the incineration dish and leave to cool. Transfer the ash to 250 ml beaker (**5.5**), moisten the ash with water, then rinse the incineration dish with total of approximately 5 ml of the hydrochloric acid (**4.1**), collecting the rinsings in the beaker; take care during the addition as a violent reaction may occur.

Evaporate the contents of the beaker to dryness on the sand bath or hot plate (**5.8**) controlled at approximately 150°C.

6.3.2 *Dissolution of Calcium*

6.3.2.1 Add to the beaker containing the test portion (*see* **6.2.2**) or the ash after incineration and rendering the silica insoluble (*see* **6.3.1**), 15 ml of the hydrochloric acid solution (**4.2**) and 120 ml of water and bring to the boil.

Filter through the filter paper (**5.4**) and collect the filtrate in a 250 ml one-mark volumetric flask (**5.6**).

6.3.2.2 If the residue on the filter shows only traces of carbon, ignore this, and wash the filter with 5 ml of the hydrochloric acid solution (**4.2**) and a little hot water, collecting the washings in the volumetric flask. Dilute to the mark with water and mix.

6.3.2.3 If the residue on the filter appears black (presence of carbon), place the filter containing the residue in a dish (**5.2**) and incinerate again in the muffle furnace, controlled at 550 ± 10°C, until all the carbonaceous matter has completely disappeared (this operation usually requires 3 to 5 h). Allow to cool, add 2 ml of the hydrochloric acid (**4.1**) and evaporate to dryness on the sand bath or hot plate (**5.8**) controlled at approximately 150°C, then add 5 ml of the hydrochloric acid solution (**4.2**). Heat, filter through a filter paper, collecting the solution in the 250 ml volumetric flask containing the filtrate previously collected (*see* **6.3.2.1**) wash the filter paper with water and dilute to the mark with water. Mix

NOTE — For the analysis of products such as alumino-calcium phosphates which are not readily soluble in hydrochloric acid, it is recommended that an alkaline fusion be carried out as follows:

Mix the sample for analysis in a platinum crucible with 5 times its mass of a mixture of equal parts of potassium carbonate and sodium carbonate. Heat carefully until the mixture has completely melted. Cool and carefully dissolve the residue in the hydrochloric acid solution as described in 6.3.2.

6.4 Blank Test

Prepare a solution in the same way as for the test solution, carrying out all the operations specified in **6.3** and using all the reagents, but omitting the test portion.

6.5 Preparation of the Calibration Graph

6.5.1 *Preparation of Calibration Solution*

Prepare a series of six 100 ml one-mark volumetric flask (**5.6**) and by means of a pipette

(**5.7**), introduce, respectively, 0 – 5 – 10—15—20 and 25 ml of the standard calcium solution (**4.4**).

Add to each flask 20 ml of the lanthanum chloride solution (**4.3**), dilute to the mark with water and mix.

These solutions correspond respectively to 0 — 2 — 4 — 6 — 8 and 10 μg of calcium per millilitre.

This series of calibration solutions is given as a guide, with the understanding that it may be displaced slightly depending on the sensitivity of the instrument. If this is the case, the operator should determine the final dilution of the test portion to obtain the optimum conditions.

NOTE — For the analysis of mineral compounds which are rich in alkali metals, it is recommended that the calibration solutions be prepared using a solution containing sodium and potassium ions in the same proportions as those of the product for analysis.

6.5.2 *Spectrometric Measurements*

Measure the absorbances of the calibration soutions (**6.5.1**), using the atomic absorption spectrometer (**5.3**), set at a wavelength of 422·7 nm, with an air/acetylene flame.

6.5.3 *Plotting the Graph*

Plot a graph having, for example, the calcium content in micrograms per millilitre, as abscissae, and the corresponding absorbances of the calibration solutions as ordinates.

6.6 Determination

6.6.1 *Preparation of Dilutions*

Carry out the dilutions according to the expected calcium content.

Using an aliquot part of the solution obtained, carry out an initial dilution using a volumetric flask, then dilute to the mark with water.

6.6.2 *Spectrometric Measurements*

Measure the absorbances of the test solution [second dilution (**6.6.1**)] and of the blank test solution (**6.4**) proceeding as described in **6.5.2**.

Correct the absorbance of the test solution if that of the blank test solution differs from that of the zero term calibration solution.

6.7 Number of Determinations

Carry out two determinations on test portions taken from the same test sample.

7 EXPRESSION OF RESULTS

The calcium content, expressed as a percentage by mass of the product as received, is equal to

$$\frac{c \times F}{40 \times m}$$

where

c = calcium content, micrograms per millilitre, of the test solution, determined from the calibration graph (**6.5.3**);

F = reciprocal dilution factor of the test solution (**6.6.1**);

m = mass; in grams, of the test portion (**6.2**).

8 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention any operations not specified in this standard, or regarded as optional, as well as any incidents likely to have affected the result.

The test report shall give all the information necessary for the complete identification of the sample.

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